UDC 678.743.22:678.01

JIS

EXHIBIT 1

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JAPANESE INDUSTRIAL STANDARD

Testing Methods for Polyvinyl Chloride

JIS K 6721 -1977

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority

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JAPANESE INDUSTRIAL STANDARD J I S

Testing Methods for Polyvinyl Chloride

K 6721-1977
(Reaffirmed: 1994)

1. Scope

This Japanese Industrial Standard specifies the testing methods for the polyvinyl chloride.

2. Definition

The polyvinyl chloride signifies polymers composed of the polyvinyl chloride as the main component.

3. Testing Methods

3.1 Specific Viscosity

3.1.1 Apparatus and Instruments The apparatus and instruments shall be as given in the following:

(1) Viscosimeter (1) Ubbelohde viscosimeter given in Fig. 1

(2) Chemical Balance Weighing capacity 100 to 200 g, reciprocal sensitivity 1 mg

(3) Desiccator The desiccator of JIS R 3503 using silica gel or calcium chloride as the desiccating agent

(4) Weighing Bottle The 50 mm flat-formed weighing bottle of JIS R 3503

(5) Measuring flask The 50 ml measuring flask of JIS R 3503

(6) Stopwatch A stopwatch graduated in 0.2 sec.

(7) Thermostatic water tank

Note (1) The Ubbelohde viscosimeter shall, as a rule, be used, however, a viscosimeter of any other type mey be used, provided that the omission of correction on the kinetic energy is allowed as the capillary-tube dimensions and the volume of test solution are equal thereto.

Applicable Standards:

JIS K 8723-p-Nytrobenzene

JIS R 3503-Glass Apparatus for Chemical Analysis

JIS Z 8401-Rules for Rounding off of Numerical Values

3.1.2 Reagents The reagents shall be as given in the following:

(1) Nytrobenzene (2) Guaranteed Grade of JIS K 8723

Note (2) That of Extra Pure Grade, after it has been purified by drying with silica gel or calcium chloride and by vacuum distillation, may be used.

3.1.3 Procedure Weigh out 200 ± 1 mg of the sample which has been dried at ordinary temperature by the chemical balance, transfer into a measuring flask, and heat to about 100 °C adding about 40 ml of the nitrobenzene. Cool when the sample has dissolued completely in appearance, further add nitrobenzene to make the total quantity 50 ml at 30 ± 0.05 °C, and consider this as test solution.

Next, pour the test solution into bulb A of the viscosimeter so that its liquid surface comes between the two marked lines. Support the viscosimeter vertically in the themostatic water tank held at 30 ± 0.05 °C, and immerse it in the tank so that the bulb C comes below the liquid surface. When the temperature of the test solution has reached the measuring temperature, close the tube 3 with a finger tip or stop the rubber tube attached to the tube with a pinch cock or the like to close up the tube completely. Next, suck up through the rubber tube being attached to the tube 2, and after the test solution has been sucked up above the upper marked line of the bulb B, release the openings of the tubes 2 and 3. Measure flow-down time in seconds when the liquid surface of the test solution passes through from the upper marked line of the bulb B down to its lower marked line.

Measure the flow-down time in seconds of the nitrobenzene in the same manner as above, and obtain the specific viscosity to three places of decimals from the following equation. Carry out three times of measurements, and take the mean value thereof.

$$\eta_{sp} = \frac{t_2}{t_1} - 1$$

where η_{sp} : specific viscosity

t_1 : flow-down time in seconds of the nitrobenzene (s)

t_2 : flow-down time in seconds of the test solution (s)

Remark: Calculation of Mean Polymerization Degree In calculating the mean polymerization degree, obtain the limiting viscosity from the equation (1), and calculate the mean polymerization degree from the equation (2):

$$[\eta] = \frac{\sqrt{2}}{C} \cdot \sqrt{\eta_{sp} - \log_e \eta_{rel}} \quad \cdots\cdots (1)$$

where $[\eta]$: limiting viscosity

η_{rel} : relative viscosity $\left(\frac{t_2}{t_1}\right)$

η_{sp} : specific viscosity

C : concentration (g/l)

$$P = 500\left\{\text{antilog}_{10}\frac{[\eta]}{0.168} - 1\right\} \quad \cdots\cdots (2)$$

where P : mean polymerization degree

Reference

The relationship between the specific viseosity and the mean polymerization degree is as given in Reference Table. An outline of the relationship between the specific viscosity and the Fikentscher's K value which is currently used in Europe and others for indicating method of mean polymerization degree is as given in Reference Figure. Calculating method of K value shall be as given in the following:

$$\log Z = \left(\frac{75k^2}{1+1.5kC} + k\right) \cdot C$$

$$K = k \cdot 10^3$$

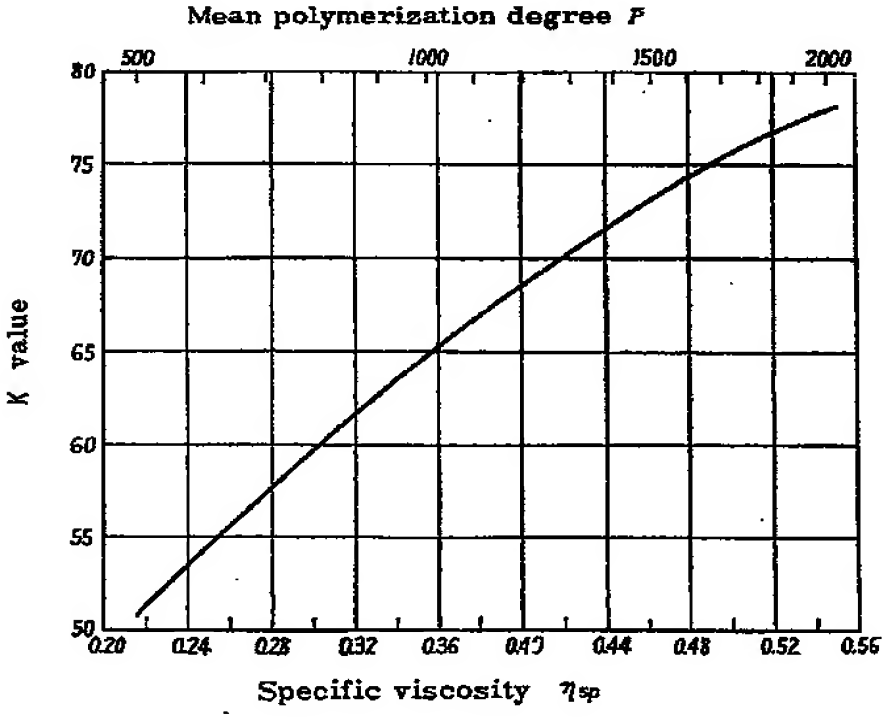
where Z : relative viscosity $\left(\frac{\text{falling down time in second (s) of test solution}}{\text{falling down time in second (s) of cyclohexanone}}\right)$

C : concentration (0.5 g/100 ml cyclohexanone)

Provided that Viscosimeter : Ubbelohde type

Measuring temperature : 25 ± 0.05°C

Reference Figure Relationship Between Specific Viscosity (η_{sp}) and Mean Polymerization Degree (P) of PVC Resin and K Value



Note: K value is measured in accordance with DIN 53726.

Reference Table Relationship Between Specific Viscosity and Mean Polymerization Degree

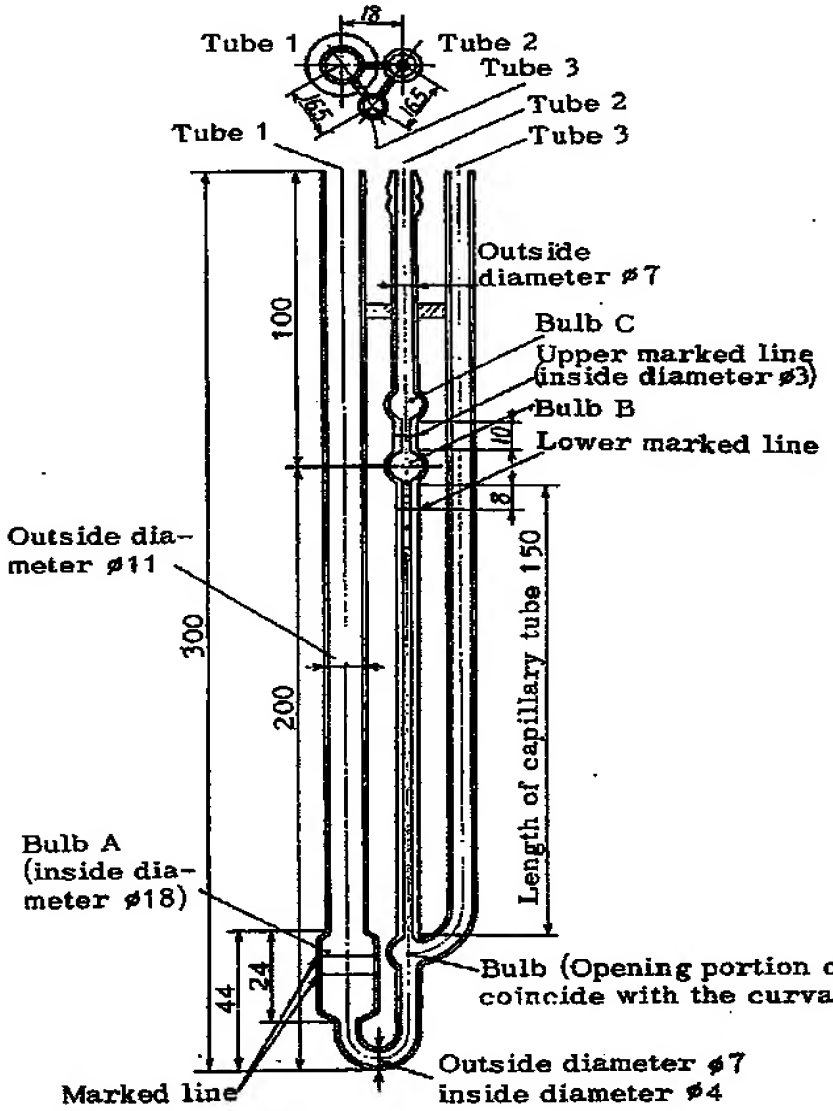
Concentration of solution 4 g/l

η_{sp}	$\bar{P}$	η_{sp}	$\bar{P}$	η_{sp}	$\bar{P}$	η_{sp}	$\bar{P}$
0.150	320	0.290	740	0.430	1340	0.570	2160
0.160	340	0.300	780	0.440	1390	0.580	2230
0.170	370	0.310	820	0.450	1440	0.590	2290
0.180	390	0.320	850	0.460	1500	0.600	2370
0.190	420	0.330	890	0.470	1560	0.610	2440
0.200	450	0.340	940	0.480	1600	0.620	2520
0.210	480	0.350	980	0.490	1660	0.630	2590
0.220	510	0.360	1020	0.500	1720	0.640	2670
0.230	540	0.370	1060	0.510	1770	0.650	2750
0.240	570	0.380	1100	0.520	1840	0.660	2830
0.250	610	0.390	1150	0.530	1900	0.670	2920
0.260	640	0.400	1200	0.540	1960	0.680	3000
0.270	670	0.410	1250	0.550	2020	0.690	3080
0.280	710	0.420	1290	0.560	2090	0.700	3170

Remark: Values of the polymerization degree are reckoned as one fraction of more than 0.5 inclusive at the end place.

Fig. 1 Ubbelohde Type Viscosimeter

unit: mm



Outside diameter of the tube 1 ø11

Outside diameter of the tube 2 ø7

Inside diameter of the capillary tube ø0.52 ± 0.04

Length of the capillary tube 150 ± 5

Volume of the bulb B 1.3 ± 0.2 ml

Remark: Figure of the upper and lower parts indicates the sketch of a plan and elevation of the Ubbelohde type viscosimeter respectively. Furthermore, no correct dimension of the tube 3 is not required.

3.2 Volatile Matter

3.2.1 Apparatus and Instruments The apparatus and instruments shall be as given in the following:

(1) Weighing Bottle A 50 mm flat-formed weighing bottle, specified in JIS R 3503

(2) Chemical Balance Weighing capacity 100 to 200 g, reciprocal sensitivity 1 mg

(3) Desiccator A desiccator specified in JIS R 3503 using silica gel or calcium chloride as desiccating agent

(4) Drier

3.2.2 Procedure Weigh out about 1 g of the sample correctly to the nearest 0.1 mg with the chemical balance, spread uniformly, and after this has been heated for 1 h at 105 ± 2 °C, cool to ordinary temperature in the desiccator, and weigh the mass. Obtain the volatile matter down to two places of decimals from the following equation. Carry out three times of measurements, and take the mean value.

$$V = \frac{B-C}{B-A} \times 100$$

where V: volatile matter (%)

A: mass of the weighing bottle (g)

B: mass of the weighing bottle containing the sample (g)

C: mass of the weighing bottle containing the sample after heating and cooling (g)

3.3 Bulk Specific Gravity

3.3.1 Apparatus and Instruments The apparatus and instruments shall be as given in the following:

(1) Measuring Apparatus for Bulk Specific Gravity The measuring apparatus for bulk specific gravity given in Fig. 2

3.3.2 Procedure After approximately 120 ml of well mixed sample has been contained in the funnel into which the damper of the measuring apparatus for bulk specific gravity is inserted, pull out the damper rapidly and drop the sample into the receiver. After the heaped up sample has been scrapped off with a glass rod, weigh the mass of the receiver containing the sample correctly to 0.1 g, and obtain the bulk specific gravity to two places of decimals from the following equation. Take the mean value by carrying out three times of measurements.

$$S_s = \frac{C-A}{B}$$

where S_s: bulk specific gravity

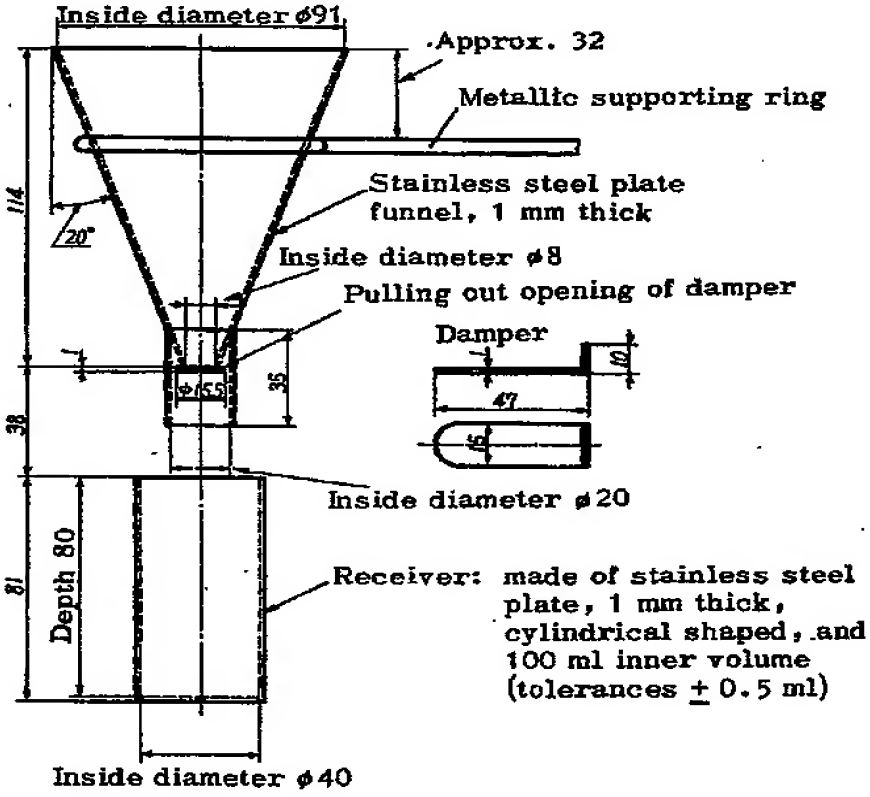
A: mass of the receiver (g)

B: inner volume of the receiver (ml)

C: mass of the receiver containing the sample (g)

Fig. 2 Measuring Instrument of Bulk Specific Gravity

unit: mm



4. Rounding off of Numerical Values for Test Results

The numerical values of the test results shall be obtained to one place bellow the specified values, and be rounded off in accordance with JIS Z 8401.

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